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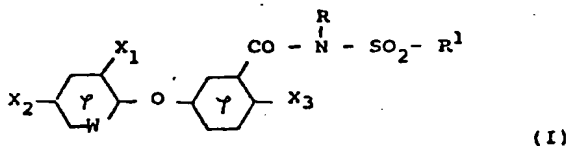
(71) Applicants  
Rhone-Poulenc  
Agrochimie,  
(France),  
14-20 rue Pierre Balzet,  
Lyon 9e,  
France.

(72) Inventors  
Alain Chene,  
Stanley T. D. Gough,  
Guy Borrod,  
Guang-Huei Lee,  
El-Ahmadi I. Heiba.

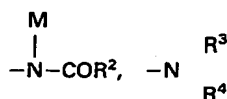
(74) Agents  
J. A. Kemp,  
14 South Square,  
Grays Inn,  
London WC1R 5EU.

(54) **Herbicidal compounds derived from N-sulphonyl-aryloxybenamides and the processes for their preparation and use**

(57) The invention provides herbicidal compounds, useful in soya and cereal crops, of the formula:



in which: W is a group  $-\text{CH}=\text{O}$  or  $-\text{CX}_4=\text{O}$  or the nitrogen atom,  $-\text{N}=\text{O}$ ;  $\text{X}_1$ ,  $\text{X}_2$ ,  $\text{X}_3$  and  $\text{X}_4$  represent a halogen atom; a polyhalogenoalkyl group;  $\text{NO}_2$ ;  $\text{CN}$ ; or an alkyl, alkoxy, alkylsulphonyl, sulphonamide, nitroso or alkoxybenzoyl radical;  $\text{R}_1$  represents a group  $-\text{OM}$ ,



or  $-\text{X}-\text{R}^5$ ; M represents a hydrogen atom or a cation which is acceptable in agriculture; X is O or S;  $\text{R}^2$  and  $\text{R}^5$  represent a substituted or unsubstituted aliphatic, cycloaliphatic or aryl hydrocarbyl group or a substituted or unsubstituted heterocyclic group;  $\text{R}^3$  and  $\text{R}^4$  have one of the meanings given for  $\text{R}^2$  and can also represent the hydrogen atom or an alkoxy radical, or can together form a single, optionally substituted divalent hydrocarbon radical; and R has one of the meanings given for M and can also represent an alkyl group having 1 to 4 C.

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## SPECIFICATION

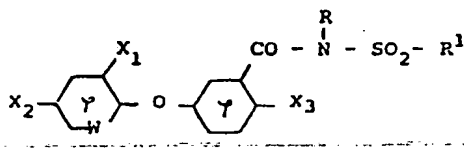
## Herbicidal compounds derived from phenoxybenzoic acids and the processes for their preparation and use

5 The present invention relates to new selective herbicides of the phenoxybenzoic acid family, to the process 5  
for their preparation and to their application.

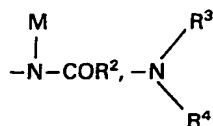
Various herbicidal derivatives of acifluorfen, which is also called 5-[2-chloro-4-(trifluoromethyl)-phenoxy]-  
2-nitrobenzoic acid, and of its salts, and in particular derivatives of the type comprising alkyl, cycloalkyl,  
thioalkyl and phenyl esters and acid amides or chlorides, are known. Such compounds are described in U.S.  
10 Patents 3,652,645, 3,784,635, 3,873,302, 3,983,168, 3,907,866, 3,798,276, 3,928,416 and 4,063,929. 10

One object of the present invention is to provide new herbicidal compounds. In fact, it is desirable to have  
available the greatest possible number of herbicides of the family in question, so as to be able to solve the  
maximum number of possible problems of weed destruction.

The present invention therefore relates to phenoxybenzoic acid derivatives of the formula:



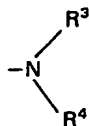
in which: W is a group  $-\text{CX}_4=$  or the nitrogen atom,  $-\text{N}=$  or, preferably, a group  $-\text{CH}=$ ;  $\text{X}_1$ ,  $\text{X}_2$ ,  $\text{X}_3$  and  $\text{X}_4$ ,  
which may be the same or different, each represent a halogen atom, in particular F, Cl and Br; a  
25 polyhalogenoalkyl group, such as  $\text{CF}_3$ ;  $\text{NO}_2$ ;  $\text{CN}$ ; or an alkyl, alkoxy, alkylsulphonyl, sulphonamide, nitroso 25  
or alkoxy carbonyl radical, the various alkyl or alkoxy groups mentioned above preferably having 1 to 4  
carbon atoms;  $\text{R}_1$  represents a group  $-\text{OM}$ ,



or  $-\text{X}-\text{R}^5$ ; M represents a hydrogen atom or a cation which is acceptable in agriculture, such as an alkali  
metal cation, in particular a sodium cation, or a substituted or unsubstituted ammonium cation; X is S or,  
preferably, O;  $\text{R}^2$  and  $\text{R}^5$  represent a substituted or unsubstituted hydrocarbonyl, i.e. aliphatic, cycloaliphatic or  
40 aryl, group, preferably having from 1 to 12 carbon atoms, or a substituted or unsubstituted heterocyclic 40  
group preferably having from 5 to 7 ring atoms;  $\text{R}^3$  and  $\text{R}^4$ , which may be same or different, each have one of  
the meanings given for  $\text{R}^2$  and can also represent the hydrogen atom or an alkoxy radical, preferably having  
from 1 to 4 carbon atoms, or can together form a single, optionally substituted divalent hydrocarbon chain  
preferably containing 4 or 5 carbon atoms in the chain; and R has one of the meanings given for M and can  
45 also represent an alkyl group having from 1 to 4 carbon atoms. 45

More preferably,  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$ , which are identical or different, represent an alkyl group preferably  
having from 1 to 4 carbon atoms, which is unsubstituted or substituted by at most 4 substituents, such as an  
aryl group (particularly a phenyl group), a chlorine or bromine atom or a hydroxy, alkoxy, SH, alkylthio,  
carboxy, alkoxy carbonyl or cyano group; an alkenyl or alkynyl group preferably having from 2 to 6 carbon  
50 atoms; or an aryl group, particularly a phenyl group, which is optionally substituted by at most 4 50  
substituents, such as Cl, Br or an alkyl, cyano, carboxy, alkoxy carbonyl,  $\text{NO}_2$ , OH, alkoxy, SH or alkylthio  
group, the various alkyl or alkoxy groups mentioned above as substituents preferably having from 1 to 4  
carbon atoms;  $\text{R}^3$  and  $\text{R}^4$  may represent the hydrogen atom or may together form an alkylene chain  
preferably having 4 or 5 carbon atoms.

55 Preferably R represents a group 55



65 or  $-\text{X}-\text{R}^5$ : compounds in which  $\text{R}^3$  represents a hydrogen atom, an unsubstituted alkyl group containing 65

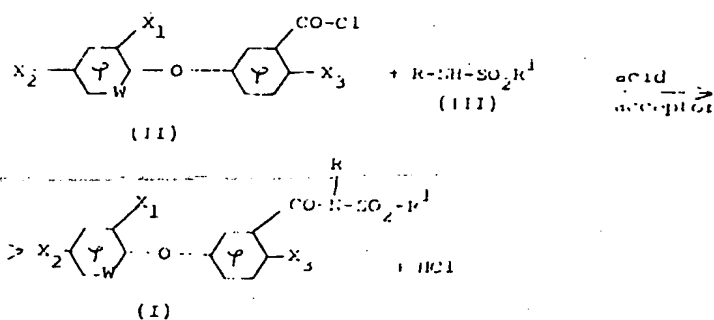
from 1 to 4 carbon atoms, e.g. methyl, an unsubstituted phenyl group or a phenyl group substituted by one or two chlorine atoms and  $R^4$  represents an unsubstituted alkyl group containing from 1 to 4 carbon atoms, an alkoxy group containing from 1 to 4 carbon atoms, or, preferably a hydrogen atom, and compounds in which X represents oxygen and  $R^5$  represents an unsubstituted alkyl group containing from 1 to 4 carbon atoms, are especially preferred.

A preferred sub-family of the compounds of the formula (I) is represented by the compounds in which  $X_1$  is the chlorine atom,  $X_2$  is  $CF_3$ ,  $X_3$  is Cl, or, preferably,  $NO_2$ , W is  $-CH=$  and R represents a hydrogen atom or a cation which is acceptable in agriculture, preferably the sodium cation.

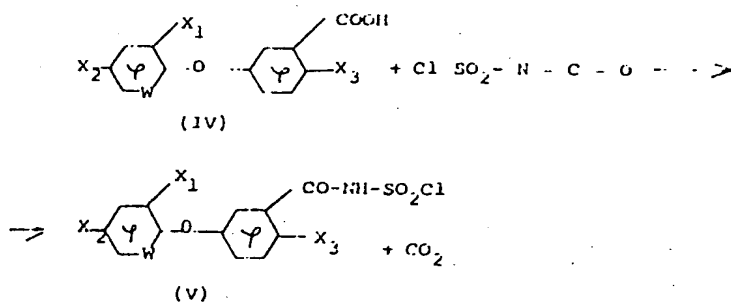
The invention also relates to the processes for the preparation of the products according to the invention.

10 These processes comprise carrying out a chemical reaction in accordance with one or other of the following reaction schemes, in which the various symbols have the meanings indicated above, unless stated otherwise.

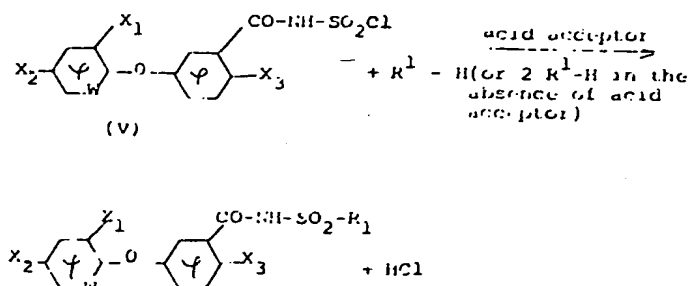
#### Scheme (I)



#### Scheme (II)

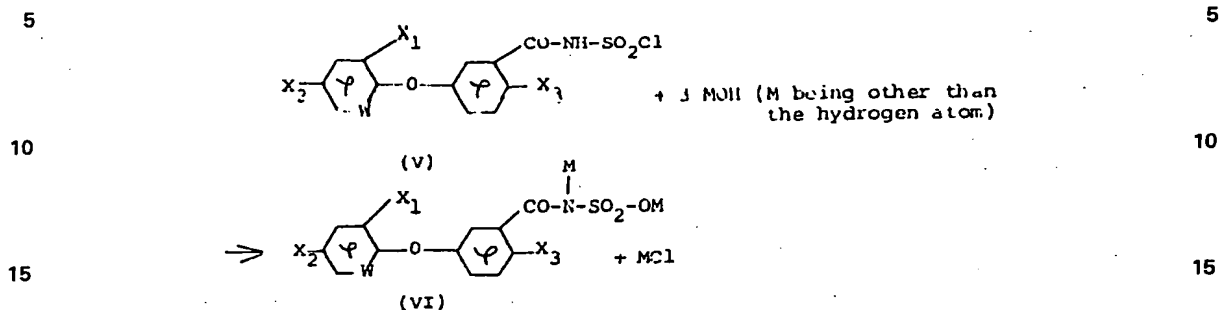


then



(Compounds of the formula (I) in which R is the hydrogen atom.)

Scheme (III) (particular case of scheme (II))



According to a feature of the present invention the phenoxybenzoic acid derivatives of general formula I may be prepared by reacting a compound of the formula (II), wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as hereinbefore defined, with a compound of the formula (III), wherein  $R$  and  $R^1$  are as hereinbefore defined, in the presence of an acid acceptor.

According to a further feature of the invention for the preparation of a compound of the formula (I) in which  $R$  represents a hydrogen atom, an acid of the formula (IV), wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as hereinbefore defined, is reacted with chlorosulphonyl isocyanate to give a product of the formula (V), wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as hereinbefore defined, and the compound of the formula (V) is then reacted with a compound of the formula  $R^1-H$ , wherein  $R^1$  is as hereinbefore defined. This process is illustrated in Scheme (II) above.

In the process according to reaction scheme (II), the first step is to react a product of the formula (IV) with chlorosulphonyl isocyanate: the reaction may be carried out in a large excess of chlorosulphonyl isocyanate or, preferably, in a solvent. The temperature is preferably from 20 to 100°C, and most preferably from 40 to 80°C; the concentration of products of the formulae (IV) and (V) is preferably from 5% to 70% by weight. If a solvent is used, it can be e.g. an optionally halogenated aliphatic or aromatic hydrocarbon, in particular toluene, or a nitrile or an ether.

The reaction of the compound of the formula (V) with the product of the formula  $R^1-H$  is preferably carried out in a solvent at a temperature from -40°C to +60°C, and most preferably from -20°C to +30°C. The solvents which can be used are the same as those of the preceding reaction step. The concentration of products of the formulae (V) and (I) is preferably from 5% to 50% by weight. The reaction is advantageously carried out in the presence of tertiary amines, in particular triethylamine and pyridine; instead of a customary acid acceptor, it is possible to use the compound  $R^1-H$  itself, which is then used in a very much larger amount than that normally used if it is simply a reactant.

According to a further feature of the present invention, for the preparation of a compound of the formula (I) in which  $R^1$  represents a group  $-OM$  and  $R$  has one of the meanings given for  $M$ ,  $M$  being as hereinbefore defined, a compound of the formula (V) wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as hereinbefore defined, is reacted with a compound of the formula  $MOH$ , wherein  $M$  is as hereinbefore defined.

The starting materials for the processes of the invention are either known compounds or can be prepared from known compounds by the application or adaptation of known methods.

It is to be understood that in this specification, unless otherwise indicated, percentages are by weight.

By the expression "known methods" as used in this specification is meant methods heretofore used or described in the chemical literature.

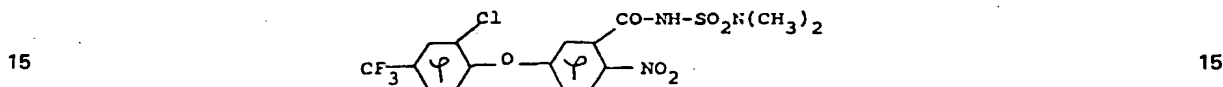
The following Examples illustrate the invention.

**Examples 1 to 19**

Acifluorfen (18.1 g) is added to chlorosulphonyl isocyanate (20 cc) and the reaction mixture is then heated gradually to 50°C, with stirring, until the evolution of HCl gas has ended. The mixture is cooled, hexane (50 cc) is added and the precipitate is filtered off, washed with hexane and then dried in vacuo. This gives

5 5-[2-chloro-4-(trifluoromethyl)-phenoxy]-2-nitro-N-chlorosulphonylbenzamide (22.2 g), which is dissolved in acetonitrile (100 cc). The solution is stirred and cooled to 0°C and a solution of dimethylamine (4.4 g) in acetonitrile (20 cc) is added dropwise. The reaction medium is then allowed to return to 20°C and the stirring is then maintained for 1 hour at this temperature. The acetonitrile is evaporated off and the residue is dissolved in chloroform. This chloroform solution is washed with water, dried and concentrated and the

10 residue is purified by chromatography on silica, chloroform being used as the eluent. This gives compound No. 4 (10.8 g) of the formula:



The various compounds Nos. 1 to 3 and 5 to 19 are prepared in the same manner, the dimethylamine being replaced by the appropriate amine or alcohol. The formulae and physical characteristics of these products are collated in Table (I) and their names are indicated below.

**Compound No.**

	1	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-ethoxysulphonylbenzamide	
25	2	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-isopropoxysulphonylbenzamide	25
	3	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-methoxysulphonylbenzamide	
	4	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N,N-dimethylaminosulphonylbenzamide	
30	5	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-methylaminosulphonylbenzamide	30
	6	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-pyrrolidin-1-ylsulphonylbenzamide	
	7	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-piperidin-1-ylsulphonylbenzamide	
	8	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(N-methoxy-N-methylamino)sulphonylbenzamide	
35	9	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-aminosulphonylbenzamide	35
	10	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-phenylaminosulphonylbenzamide	
	11	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(4-chlorophenyl)aminosulphonylbenzamide	
	12	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(3-chlorophenyl)aminosulphonylbenzamide	
40	13	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2-chlorophenyl)aminosulphonylbenzamide	40
	14	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,6-dichlorophenyl)aminosulphonylbenzamide	
	15	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,3-dichlorophenyl)aminosulphonylbenzamide	
45	16	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(3,4-dichlorophenyl)aminosulphonylbenzamide	45
	17	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,5-dichlorophenyl)aminosulphonylbenzamide	
50	18	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,4-dichlorophenyl)aminosulphonylbenzamide	50
	19	: 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(3,5-dichlorophenyl)aminosulphonylbenzamide.	

**55 Example 20: Herbicidal application in the pre-emergence treatment of plant species**

55

A number of seeds are sown in 9 x 9 x 9 cm pots filled with light agricultural earth, this number being determined as a function of the plant species and the size of the seed.

The pots are treated by spraying with an amount of spraying mixture which corresponds to a volumetric application dose of 500 l/ha and contains the active ingredient at the desired concentration.

60 The treatment with the spraying mixture is therefore carried out on seeds not covered with earth (the term "spraying mixture" is used to denote, in general, compositions diluted with water, such as they are applied to the plants).

60

The spraying mixture used for the treatment is an aqueous suspension of the active ingredient containing 0.1% by weight of Cemulsol NP10 (a surface-active agent consisting of an ethylene oxide/alkylphenol

65 condensate, mainly of a 10:1 ethylene oxide/nonylphenol condensate) and 0.04% by weight of Tween 20 (a

65

surface-active agent consisting of the laurate of an ethylene oxide/sorbitol condensate).

This suspension was obtained by mixing and grinding the ingredients in a microniser so as to give an average particle size of less than 40 microns.

According to the concentration of active ingredient in the spraying mixture, the dose of active ingredient applied was 0.25 to 4 kg/ha.

After treatment, the seeds are covered with an approximately 3 mm thick layer of earth.

The pots are then placed in troughs which are intended to receive the moistening water, for sub-irrigation, and are kept for 21 days at a temperature of 22-24°C and under 70% relative humidity.

After 21 days, the number of living plants in the pots treated with the spraying mixture containing the active ingredient to be tested, and the number of living plants in a control pot treated under the same conditions, but by means of a spraying mixture not containing active ingredient, are counted. The percentage destruction of the treated plants, relative to the untreated control, is thus determined. A percentage destruction equal to 100% indicates that there has been complete destruction of the plant species in question, and a percentage of 0% indicates that the number of living plants in the treated pot is identical to that in the control pot.

*Example 21: Herbicidal application in the post-emergence treatment of plant species*

A number of seeds are sown in 9 x 9 x 9 cm pots filled with light agricultural earth, this number being determined as a function of the plant species and the size of the seed.

The seeds are then covered with an approximately 3 mm thick layer of earth and the seed is left to germinate until it has produced a plantlet at the appropriate stage of development. The treatment stage for the graminaceous plants is the stage of "second leaf forming". The appropriate stage for soya is the stage of "first trifoliate leaf open". The treatment stage for the other dicotyledons is the stage of "cotyledons open, first true leaf developing".

The pots are then treated by spraying with an amount of spraying mixture which corresponds to a volumetric application dose of 500 l/ha and contains the active ingredient at the desired concentration.

The spraying mixture was prepared in the same manner as in Example 20.

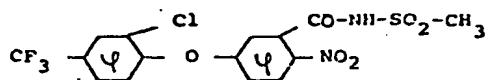
According to the concentration of active ingredient in the spraying mixture, the dose of active ingredient applied was 0.25 to 4 kg/ha.

The treated pots are then placed in troughs which are intended to receive the moistening water, for sub-irrigation, and are kept for 21 days at a temperature of 22-24°C and under 70% relative humidity.

After 21 days, the number of living plants in the pots treated with the spraying mixture containing the active ingredient to be tested, and the number of living plants in a control pot treated under the same conditions, but by means of a spraying mixture not containing active ingredient, are counted. The percentage destruction of the treated plants, relative to the untreated control, is thus determined. A percentage destruction equal to 100% indicates that there has been complete destruction of the plant species in question, and a percentage of 0% indicates that the number of living plants in the treated pot is identical to that in the control pot.

The plant species on which the herbicidal applications were carried out are indicated in Table (II).

The results obtained in Examples 20 and 21 have been indicated in Table (III), which collates both the pre-emergence results according to Example 20 and the post-emergence results according to Example 21. In the course of these experiments, it was possible to observe improved performance characteristics (in particular the selectivity) compared with a product representative of the prior art (European Patents 3,416 and 23,392) and having the formula:



*Example 22:*

Excellent herbicidal results are also obtained using the sodium salts (compounds containing the N-Na group) of compounds Nos. 4, 5 and 9.

The experiments carried out therefore show the notably advantageous properties of the compounds according to the invention, both when used for the pre-emergence treatment of crops, more particularly of soya, and for the post-emergence treatment, in particular of soya and cereals, including maize. In the case of soya, the activity of the compounds is particularly advantageous if this crop is infested with dicotyledon weeds such as abutilon, xanthium and ipomea. The products of the invention show an improved selectivity compared with the reference product.

For their use in practice, the compounds according to the invention are rarely employed by themselves. Most frequently, the compounds form part of compositions. The present invention provides herbicidal compositions which comprise, as active ingredient, a compound of formula (I) in association with a herbicidally acceptable carrier. The carrier may be solid or liquid. The compositions may also comprise herbicidally acceptable surface-active agents. In particular, the customary inert carriers and the customary surface-active agents can be used.

These compositions can also contain various kinds of other ingredients, such as e.g. protective colloids, adhesives, thickeners, thixotropic agents, penetrating agents, stabilisers and sequestering agents, and also other known active ingredients having pesticidal properties (in particular insecticidal, fungicidal or herbicidal properties), properties promoting plant growth (in particular fertilisers) or properties regulating plant growth. More generally, the compounds used in the invention can be combined with any of the solid or liquid additives corresponding to the usual formulation techniques.

The use doses of the compounds used in the invention can vary within wide limits, in particular according to the nature of the adventitious plants to be removed and the usual degree of infestation of the crops by these adventitious plants.

10 In general, the compositions according to the invention usually contain from 0.05 to about 95% (by weight) of one or more active ingredients according to the invention, from 1% to about 95% of one or more solid or liquid carriers and, if appropriate, from 0.1 to about 20% of one or more surface-active agents.

According to what has already been stated, the compounds used in the invention are generally combined with carriers and, if appropriate, surface-active agents.

15 In the present account, the term "carrier" denotes an organic or inorganic, natural or synthetic material with which the active ingredient is combined in order to facilitate its application to the plant, to seeds or to the soil. This carrier is therefore generally inert and it must be acceptable in agriculture, in particular on the plant treated. The carrier can be solid (e.g. clays, natural or synthetic silicates, silica, resins, waxes and solid fertilisers) or liquid (e.g. water, alcohols, in particular butanol, esters, in particular methylglycol acetate, ketones, in particular cyclohexanone and isophorone, petroleum fractions, aromatic hydrocarbons, in particular xylenes, or paraffinic hydrocarbons, aliphatic chlorohydrocarbons, in particular trichloroethane, or aromatic chlorohydrocarbons, in particular chlorobenzenes, water-soluble solvents, such as dimethylformamide, dimethyl sulphoxide and N-methylpyrrolidone, and liquefied gases).

The surface-active agent can be an emulsifying, dispersing or wetting agent of ionic or non-ionic type or a mixture of such surface-active agents. Examples which may be mentioned are salts of polyacrylic acids, salts of lignosulphonic acids, salts of phenolsulphonic or naphthalenesulphonic acids, polycondensates of ethylene oxide with fatty alcohols, fatty acids or fatty amines, substituted phenols (in particular alkylphenols or arylphenols), salts or sulposuccinic acid esters, taurine derivatives (in particular alkyltaurates), phosphoric acid esters of condensates of ethylene oxide with alcohols or phenols, fatty acid esters of polyols, and derivatives of the above compounds containing sulphate, sulphonate and phosphate groups. The presence of at least one surface-active agent is generally essential if the active ingredient and/or the inert carrier are not soluble in water and if the vehicle of application is water.

For their application, the compounds of the formula (I) are therefore generally in the form of compositions; these compositions according to the invention are themselves in a fairly wide variety of solid or liquid forms.

35 As forms of solid compositions, there may be mentioned dusting powders (with a content of compound of the formula (I) which can range up to 100%) and granules, in particular those obtained by extrusion, by compaction, by the impregnation of a granular carrier or by the formation of granules from a powder (the content of compound of the formula (I) in these granules being from 0.5 to 80% for the latter cases). The solid compositions most frequently contain 20 to 80% of active ingredient.

40 As forms of liquid compositions or compositions which are to be made up into liquid compositions on application, there may be mentioned solutions, in particular emulsifiable concentrates, emulsions, suspension concentrates, aerosols, wettable powders (or spraying powders), dry flowables and pastes.

The liquid compositions most frequently contain 10 to 80% of active ingredient.

45 The emulsifiable or soluble concentrates most frequently comprise 10 to 80% of active ingredient, and the emulsions or solutions which are ready for application contain 0.01 to 20% of active ingredient. In addition to the solvent, the emulsifiable concentrates can contain, where necessary, 2 to 20% of suitable additives, such as stabilisers, surface-active agents, penetrating agents, corrosion inhibitors, dyestuffs and adhesives.

Starting from these concentrates, emulsions or solutions of any desired concentration, which are particularly suitable for application to the plants, can be obtained by dilution with water.

50 The compositions of a few emulsifiable concentrates are now given as examples:

#### Example 23

55	active ingredient	250 g	55
	ethylene oxide/alkylphenol condensate	30 g	
	calcium alkylarylsulphonate	50 g	
60	petroleum distillation cut distilling at between 160 and 185°C	670 g	60

Another formulation is:

*Example 24*

5	active ingredient	350 g	5
	ethylene oxide/caster oil condensate	60 g	
	sodium alkylarylsulphonate	40 g	
10	cyclohexanone	150 g	10
	xylene	400 g	

Another formulation is:

15 *Example 25*

15

	active ingredient	400 g	
20	ethylene oxide/alkylphenol condensate	100 g	20
	ethylene glycol methyl ether	250 g	
25	aromatic petroleum cut distilling at between 160 and 185°C	250 g	25

Another formulation is:

*Example 26*

30	active ingredient	400 g	30
	phosphate of ethylene oxide/tristyrylphenol condensate	50 g	
35	phosphate of ethylene oxide/alkylphenol condensate	65 g	35
	sodium alkylbenzenesulphonate	35 g	
40	cyclohexanone	300 g	40
	aromatic petroleum cut distilling at between 160 and 185°C	150 g	

Another formulation is:

45 *Example 27*

45

	active ingredient	400 g/l	
50	alkali metal dodecylbenzenesulphonate	24 g/l	50
	10:1 ethylene oxide/nonylphenol condensate	16 g/l	
55	cyclohexanone	200 g/l	55
	aromatic solvent	q.s.p. 1 litre	



Another formulation of an emulsifiable concentrate uses the following:  
*Example 28*

5	active ingredient	250 g	5
	epoxidised vegetable oil	25 g	
10	mixture of an alkylarylsulphonate and a polyglycol ether of fatty alcohols	100 g	10
	dimethylformamide	50 g	
	xylene	575 g	

15 The suspension concentrates, which can be applied by spraying, are prepared so as to give a stable fluid product which does not form a deposit (by fine grinding), and they usually contain from 10 to 75% of active ingredient, from 0.5 to 15% of surface-active agents, from 0.1 to 10% of thixotropic agents, from 0 to 10% of suitable additives, such as anti-foam agents, corrosion inhibitors, stabilisers, penetrating agents and adhesives, and, as the carrier, water or an organic liquid in which the active ingredient is sparingly soluble or insoluble; certain organic solids, or inorganic salts, can be dissolved in the carrier in order to assist in preventing sedimentation or to act as anti-freeze agents for the water.

A composition of a suspension concentrate is now given as an example:

*Example 29*

25	active ingredient	50 g	25
	phosphate of ethylene oxide/tristyryl-phenol condensate	50 g	
30	ethylene oxide/alkylphenol condensate	50 g	30
	sodium polycarboxylate	20 g	
35	ethylene glycol	50 g	35
	organopolysiloxane oil (anti-foam agent)	1 g	
40	polysaccharide	12.5 g	40
	water	316.5 g	

The wettable powders (or spraying powders) are usually prepared so as to contain 20 to 95% of active ingredient, and they usually contain, in addition to the solid carrier, from 0 to 5% of a wetting agent, from 3 to 10% of a dispersing agent and, where necessary, from 0 to 10% of one or more stabilisers and/or other additives, such as penetrating agents, adhesives, anti-caking agents, and dyestuffs.

Various compositions of wettable powders are now given as examples:

50	<i>Example 30</i>		50
	active ingredient	50%	
55	calcium lignosulphonate (defluoculant)	5%	55
	isopropyl-naphthalenesulphonate (anionic wetting agent)	1%	
60	anti-caking silica	5%	60
	kaolin (filler)	39%	

Another example of a wettable powder, this time of 80% strength, is given below:

*Example 31*

5	active ingredient	80%	5
	sodium alkylnaphthalenesulphonate	2%	
	sodium lignosulphonate	2%	
10	anti-caking silica	3%	10
	kaolin	13%	

15 Another example of a wettable powder is given below: 15

*Example 32*

20	active ingredient	50%	20
	sodium alkylnaphthalenesulphonate	2%	
	low-viscosity methylcellulose	2%	
25	diatomaceous earth	46%	25

Another example of a wettable powder is given below:

*Example 33*

30	active ingredient	90%	30
	sodium dioctyl-sulphosuccinate	0.2%	
35	synthetic silica	9.8%	35

Another composition of a spraying powder, this time of 40% strength, uses the following constituents:

*Example 34*

40	active ingredient	400 g	40
	sodium lignosulphonate	50 g	
45	sodium dibutyl-naphthalenesulphonate	10 g	45
	silica	540 g	

Another composition of a spraying powder, this time of 25% strength, uses the following constituents:

50 *Example 35* 50

	active ingredient	250 g	
55	isooctylphenoxy-polyoxyethylene-ethanol	25 g	55
	mixture of equal parts by weight of Champagne chalk and hydroxyethylcellulose	17 g	
60	sodium aluminosilicate	543 g	60
	kieselguhr	165 g	

Another composition of a spraying powder, this time of 10% strength, uses the following constituents:

*Example 36*

5	active ingredient	100 g	5
	mixture of sodium salts of saturated fatty acid sulphates	30 g	
10	naphthalenesulphonic acid/formaldehyde condensate	50 g	10
	kaolin	820 g	

- 15 To obtain these spraying powders or wettable powders, the active ingredients are intimately mixed with the additional substances, in suitable mixers, or the porous filler is impregnated with the molten active ingredient, and the mixture is ground in mills or other suitable grinders. This gives spraying powders of advantageous wettability and suspendability; they can be suspended in water at any desired concentration, and this suspension can be used very advantageously, in particular for application to the leaves of plants.
- 20 The dry flowables (more exactly, these are granules which are readily dispersible in water) have a composition substantially similar to that of the wettable powders. They can be prepared by the formation of granules from formulations described for the wettable powders, either by a wet process (bringing of the finely divided active ingredient into contact with the inert filler and with a small amount of water, e.g. 1 to 20%, or of an aqueous solution of dispersing agent or binder, followed by drying and sieving) or by a dry process (compaction, followed by grinding and sieving).
- 25 A formulation of a dry flowable is now given as an example:

*Example 37*

30	active ingredient	800 g	30
	sodium alkyl naphthalenesulphonate	20 g	
35	methylene-bis-(sodium naphthalene-sulphonate)	80 g	35
	kaolin	100 g	

- In place of the wettable powders, it is possible to produce pastes. The conditions and modes of production and use of these pastes are similar to those of the wettable powders or spraying powders.
- 40 As already stated, the aqueous dispersions and emulsions, e.g. compositions obtained by diluting, with water, a wettable powder or an emulsifiable concentrate according to the invention, are included within the general scope of the compositions which can be used in the present invention. The emulsions can be of the water-in-oil or oil-in-water type and they can have a thick consistency such as that of a "mayonnaise".
- 45 All these aqueous dispersions or emulsions, or spraying mixtures, can be applied to the crops in which weeds are to be destroyed, by any suitable method, mainly by spraying, at doses which are generally of the order of 100 to 1,200 litres of spraying mixture per hectare.
- The granules, which are intended to be placed on the soil, are usually prepared so that they have dimensions of between 0.1 and 2 mm, and they can be manufactured by agglomeration or impregnation.
- 50 Preferably, the granules contain 1 to 25% of active ingredient and 0 to 10% of additives, such as stabilisers, slow-release modifiers, binders and solvents.

One example of a granular composition uses the following constituents:

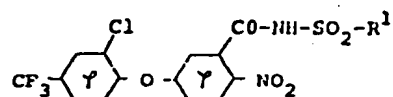
*Example 38*

55	active ingredient	50 g	55
	propylene glycol	25 g	
60	clay (particle size: 0.3 to 0.8 mm)	925 g	60

- As indicated above, the invention also relates to a method of controlling the growth of weeds at a locus used, or to be used, for growing crops, in particular cereals, such as wheat and including maize and also soya, which method comprises applying to the locus an effective amount of a compound of general formula
- 65 I. The compound of general formula I may be applied to the plants and/or to the soil in the area in which

- weeds are to be destroyed. In practice, the compounds are used in the form of herbicidal compositions according to the invention, which have been described above. In general, amounts of active ingredient ranging from 0.01 to 5 kg/ha, preferably from 0.1 to 2 kg/ha, give good results, it being understood that the choice of the amount of active ingredient to be used depends on the severity of the problem to be solved, the climatic conditions and the crop in question. The treatment can be carried out either as a pre-emergence treatment of the crops and adventitious plants, or as a pre-sowing treatment of the crops with incorporation into the soil (such incorporation constitutes a feature of the method of the invention), or as a post-emergence treatment. Other embodiments of the treatment method according to the invention can also be used: thus, it is possible to apply the active ingredient to the soil, with or without incorporation, before planting out a crop.
- The treatment method of the invention can be used for both annual crops and in the case of perennial crops; in the latter case, it is preferred to apply the active ingredient of the invention in a localised manner, e.g. between the rows of the said crops.

Table (I): products of the formula



Compound No.	R <sup>1</sup>	M.p. in °C	Yield
1	CH <sub>3</sub> CH <sub>2</sub> -O-	150	76.5 %
2		110	85 %
3	CH <sub>3</sub> -O-	150	38 %
4	(CH <sub>3</sub> ) <sub>2</sub> N-	73	48 %
5	CH <sub>3</sub> -NH-	205	27 %
6			39 %
7		192	46 %
8		140	27 %
9	NH <sub>2</sub>	135-140	59 %

Table (I) (continuation)











Compound No.	R <sup>1</sup>	-M.p.	Yield
10		188	90 %
11		190	84 %
12		190	68 %
13		183	29.5 %
14		155	34 %
15		80	79 %
16		180	87 %
17		65	25.5 %
18		180	72 %
19		185	76.5 %

TABLE II

American name		Latin name	Abbreviation
Crops	Wheat		WHE
	Soybean		SOY
Weeds	Barneyard grass	Echinochloa crus-galli	ECH
	Velvet leaf	Abutilon theophrasti	ABU
	Cocklebur	Xanthium pennsylvanicum	XAN
	Wild mustard	Sinapis arvensis	SIN
	Morning glory (annual)	Ipomea purpurea	IPO

TABLE (III)

Compound No.	Doses in kg/ha	Pre-Emergence							Post-Emergence							5
		ECH	ABU	IPO	SIN	XAN	WHE	SOY	ECH	ABU	IPO	SIN	XAN	WHE	SOY	
10	4	95	100	100	100	90	20	0	60	100	100	100	100	0	0	10
1	1	29	95	100	100	20	0	0	0	80	100	100	100	0	0	
	0,25	0	95	80	100	0	0	0	0	80	90	100	100	0	0	
15	4	90	100	100	100	100	20	0	80	100	100	100	100	0	0	15
2	1	60	100	0	100	0	0	0	30	50	100	100	100	0	0	
20	0,25	0	90	0	100	0	0	0	0	50	90	100	100	0	0	20
	4	100	100	80	100	0	0	0	100	100	100	100	100	0	30	
3	1	50	100	50	100	0	0	0	30	30	100	100	100	0	0	
25	0,25	20	100	0	100	0	0	0	0	0	100	100	100	0	0	25
	4	100	100	90	100	0	60	0	100	90	100	100	100	0	0	
30	1	100	100		100		50	0	100	80	100	100	100	0	0	30
	0,25	100	100	20	100	0	0	0	60	30	100	100	100	0	0	
	4	100	100	100	100	100	98	0	100	100	100	100	100	0	0	
35	1	100	100	100	100	0	30	0	100	100	100	100	100	0	0	35
5	0,25	90	100	50	100	0	0	0	80	0	100	100	100	0	0	
40	4	0	100	0	100	0	0	0	20	0	100	100	100	0	0	40
6	1	0	100	0	100	0	0	0	0	0	100	100	100	0	0	
	0,25	0	80	0	100	0	0	0	0	0	100	90	100	0	0	
45	4	0	60	0	100	0	0	0	0	30	100	80	100	0	0	45
7	1	0	30	0	30	0	0	0	0	30	80	60	0	0	0	
50	0,25	0	20	0	0	0	0	0	0	30	80	50	0	0	0	50
	4	100	100	80	100	30	0	0	100	100	100	100	100	0	0	
8	1	30	100	80	100	0	0	0	60	100	100	100	100	0	0	
55	0,25	0	90	30	100	0	0	0	0	100	100	100	100	0	0	55
	4	0	100	30	100	0	0	100	20	0	100	100	100	0	0	
60	1	0	100	0	100	0	0	0	0	0	80	100	100	0	0	60
	0,25	0	100	0	98	0	0	0	0	0	80	95	100	0	0	
	4	90	100	100	100	100	0	0	50	30	100	100	100	0	0	

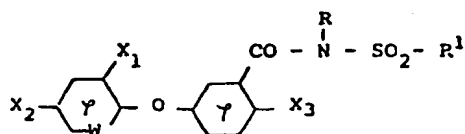
TABLE (III)

5	Compound No.	Doses in kg/ha	Pre-Emergence							Post-Emergence							5
			ECH	ABU	IPO	SIN	XAN	WHE	SOY	ECH	ABU	IPO	SIN	XAN	WHE	SOY	
10	11	1	30	98	0	100	100	0	0	20	0	100	100	100	0	0	10
		0,25	0	50	0	100	50	0	0	20	0	90	80	100	0	0	
		4	60	100	80	100	0	0	0	0	0	100	90	100	0	0	
15	12	1	0	80	80	100	0	0		0	0	80	90	100	0	0	15
		0,25	0	0	50	0	0	0	0	0	0	80	80	50	0	0	
		4	0	80	30	100	0	0	0	0	20	80	100	100	0	0	
20	13	1	0	80	0	100	0	0	0	0	0	80	90	100	0	0	20
		0,25	0	0	0	30	0	0	0	0	0	80	80	0	0	0	
		4	95	90	0	100	0	0	50	50	20	100	100	100	0	0	
25	14	1	30	90	0	100	0	0	0	30	0	100	100	100	0	0	25
		0,25	0	80	0	80	0	0	0	0	0	100	90	0	0	0	
		4	80	100	80	100	100	0	0	0	0	90	80	100	0	0	
30	15	1	0	30	50	100	100	0	0	0	0	80	80	100	0	0	30
		0,25	0	0	0	20	0	0	0	0	0	80	30	100	0	0	
		4	50	90	0	100	0	0	0	0	0	80	90	100	0	0	
40	16	1	0	0	0	100	0	0	0	0	0	60	80	100	0	0	40
		0,25	0	0	0	20	0	0	0	0	0	60	50	50	0	0	
		4	90	100	50	100	0	0	0	30	0	100	90	100	0	0	
45	17	1	30	100	0	100	0	0	100	20	0	50	80	50	0	0	45
		0,25	0	60	0	50	0	0	0	0	0	50	30	0	0	0	
		4	80	100	100	100	100	0	0	30	20	100	100	100	0	0	
50	18	1	0	80	0	100	0	0	0	0	0	80	100	50	0	0	50
		0,25	0	50	0	80	0	0	100	0	0	50	20	50	0	0	
		4	30	80	80	100	0	0	50	30	0	100	50	0	0	0	
55	19	1	0	30	0	90	0	0	0	0	0	100	100	0	0	0	55
		0,25	0	20	0	0	0	0	0	0	0	100	50	0	0	0	
		4	30	80	80	100	0	0	50	30	0	100	50	0	0	0	
60	19	1	0	30	0	90	0	0	0	0	0	100	100	0	0	0	60
		0,25	0	20	0	0	0	0	0	0	0	100	50	0	0	0	
		4	30	80	80	100	0	0	50	30	0	100	50	0	0	0	

## CLAIMS

1. A phenoxybenzoic acid derivative of the formula:

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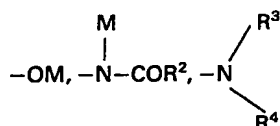
(I)

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in which: W is a group  $-\text{CH}=\text{}$  or  $-\text{CX}_4=\text{}$  or the nitrogen atom,  $-\text{N}=\text{}$ ;  $\text{X}_1$ ,  $\text{X}_2$ ,  $\text{X}_3$  and  $\text{X}_4$ , which may be the same or different, each represent a halogen atom; a polyhalogenoalkyl group;  $\text{NO}_2$ ;  $\text{CN}$ ; or an alkyl, alkoxy, alkylsulphonyl, sulphonamide, nitroso or alkoxycarbonyl radical;  $\text{R}_1$  represents a group

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or  $-\text{X}-\text{R}^5$ ; M represents a hydrogen atom or a cation which is acceptable in agriculture; X is O or S;  $\text{R}^2$  and  $\text{R}^5$  represent a substituted or unsubstituted aliphatic, cycloaliphatic or aryl hydrocarbonyl group or a substituted or unsubstituted heterocyclic group;  $\text{R}^3$  and  $\text{R}^4$ , which may be the same or different, each have one of the meanings given for  $\text{R}^2$  and can also represent the hydrogen atom or an alkoxy radical, or can together form a single, optionally substituted divalent hydrocarbon chain; and R has one of the meanings given for M and can also represent an alkyl group having 1 to 4 carbon atoms.

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2. A compound according to claim 1, in which the alkyl or alkoxy radicals which it contains have from 1 to 4 carbon atoms.

3. A compound according to claim 1 or 2 in which  $\text{X}_1$ ,  $\text{X}_2$ ,  $\text{X}_3$  and  $\text{X}_4$  represent F, Cl, Br or  $\text{CF}_3$ .

4. A compound according to claim 1, 2 or 3 in which  $\text{R}^2$  and  $\text{R}^5$  represent a substituted or unsubstituted hydrocarbonyl group containing 1 to 12 carbon atoms.

5. A compound according to claim 1, 2 or 3 in which  $\text{R}^2$  and  $\text{R}^5$  represent a substituted or unsubstituted heterocyclic group having from 5 to 7 ring atoms.

6. A compound according to any one of the preceding claims in which W represents  $-\text{CH}=\text{}$ .

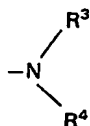
7. A compound according to any one of the preceding claims in which  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  which are identical or different represent an alkyl group which is unsubstituted or substituted by at most 4 substituents; an alkenyl or alkynyl group; or an aryl group, which is optionally substituted by at most 4 substituents.

8. A compound according to claim 7 in which  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  which are identical or different, represent an alkyl group having from 1 to 4 carbon atoms which is substituted by at most four substituents selected from an aryl group, a chlorine or bromine atom, or a hydroxy, alkoxy, SH, alkylthio, carboxy, alkoxycarbonyl or cyano group; the alkenyl and alkynyl groups have from 2 to 6 carbon atoms; and the aryl groups represented by  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  or  $\text{R}^5$  are optionally substituted by at most four substituents selected from Cl, Br, alkyl, cyano, carboxy, alkoxycarbonyl,  $\text{NO}_2$ , OH, alkoxy, SH or alkylthio groups.

9. A compound according to claim 8 in which the aryl groups are phenyl and alkyl and alkoxy radicals contain from 1 to 4 carbon atoms.

10. A compound according to any one of the preceding claims in which  $\text{R}^1$  represents a group

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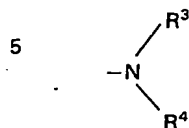
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or  $-\text{X}-\text{R}^5$  in which  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  are as defined in claim 1.



11. A compound according to any one of the preceding claims in which R<sup>1</sup> represents a group



10 in which R<sup>3</sup> represents a hydrogen atom, an unsubstituted alkyl group containing from 1 to 4 carbon atoms, an unsubstituted phenyl group or a phenyl group substituted by one or two chlorine atoms, and R<sup>4</sup> represents an unsubstituted alkyl group containing from 1 to 4 carbon atoms, an alkoxy group containing from 1 to 4 carbon atoms, or a hydrogen atom.

15 12. A compound according to claim 11 in which R<sup>4</sup> represents the hydrogen atom.

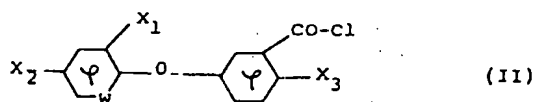
13. A compound according to any one of claims 1 to 9 in which R represents a group -X-R<sup>5</sup> in which X represents oxygen and R<sup>5</sup> represents an unsubstituted alkyl group containing from 1 to 4 carbon atoms.

14. A compound according to any one of the preceding claims in which X<sub>1</sub> is Cl, X<sub>2</sub> is CF<sub>3</sub>, W is -CH=, X<sub>3</sub> is Cl or NO<sub>2</sub> and R is a hydrogen atom or a cation which is acceptable in agriculture.

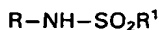
20 15. A compound according to claim 14 in which X<sub>3</sub> is NO<sub>2</sub>.

16. 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-ethoxysulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-isopropoxysulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-methoxysulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N,N-dimethylaminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-methylaminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-pyrrolidin-1-ylsulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-piperidin-1-ylsulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(N-methoxy-N-methylamino)sulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-phenylaminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(4-chlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(3-chlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2-chlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,6-dichlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,3-dichlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(3,4-dichlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,5-dichlorophenyl)aminosulphonylbenzamide, 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(2,4-dichlorophenyl)aminosulphonylbenzamide and 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitro-N-(3,5-dichlorophenyl)aminosulphonylbenzamide.

40 17. A process for the preparation of a compound according to claim 1 which comprises reacting a compound of the formula



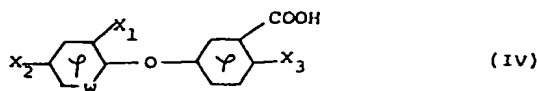
(wherein X<sub>1</sub>, X<sub>2</sub>, X<sub>3</sub> and W are as defined in claim 1) with a compound of the formula:



(III)

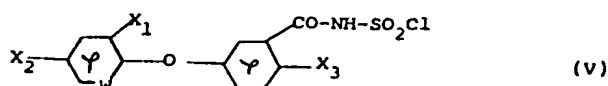
(wherein R and R<sup>1</sup> are as defined in claim 1) in the presence of an acid acceptor.

55 18. A process for the preparation of a compound according to claim 11 in which R represents a hydrogen atom, which comprises reacting an acid of the formula:



(wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as defined in claim 1) with chlorosulphonyl isocyanate to give a product of the formula:

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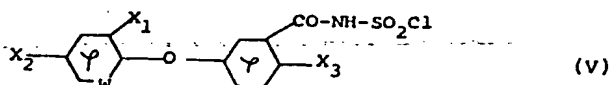
(wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as defined in claim 1) and then reacting the compound of the formula (V) with a compound of the formula  $R^1-H$ , wherein  $R^1$  is as defined in claim 1.

19. A process according to claim 18 in which the reaction of the acid of the formula (IV) with chlorosulphonyl isocyanate is carried out in a solvent, at a temperature from  $20^\circ$  to  $100^\circ\text{C}$  and with a concentration of compounds of formula (IV) and (V) of from 5% to 70% by weight.

20. A process according to claim 18 or 19 in which the reaction of the compound of formula (V) with a compound of the formula  $R^1-H$  is carried out at a temperature from  $-40^\circ\text{C}$  to  $+60^\circ\text{C}$ , in a solvent and with a concentration of compounds of the formulae (V) and (I) of from 5% to 50% by weight.

21. A process for the preparation of a compound according to claim 1 in which  $R^1$  represents a group  $-OM$  and  $R$  represents a group  $M$ ,  $M$  being as defined in claim 1, which comprises reacting a compound of the formula:

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(wherein  $X_1$ ,  $X_2$ ,  $X_3$  and  $W$  are as defined in claim 1) with a compound of the formula  $MOH$ , wherein  $M$  is as defined in claim 1.

22. A process according to any one of claims 17 to 21 substantially as hereinbefore described.

23. A process according to any one of claims 17 to 21 substantially as hereinbefore described in any one of Examples 1 to 19.

24. A compound according to claim 1 when prepared by a process as claimed in any one of claims 17 to 23.

25. A herbicidal composition which comprises, as active ingredient, a compound according to any one of claims 1 to 16 and 24 in association with a herbicidally acceptable carrier.

26. A composition according to claim 25, which contains 0.05 to 95% by weight of active ingredient.

27. A composition according to claim 25 or 26, which is liquid and which contains 10 to 80% of active ingredient.

28. A composition according to claim 25 or 26, which is solid and contains 20 to 80% of active ingredient.

29. A composition according to any one of claims 25 to 28, which contains 0.1 to 20% of surface-active agent.

30. A composition according to claim 25 substantially as hereinbefore described in any one of Examples 23 to 38.

31. A method of controlling the growth of weeds at a locus used, or to be used, for growing crops, which comprises applying to the locus an effective amount of a compound according to any one of claims 1 to 16 and 24.

32. A method according to claim 31, wherein the composition is applied to a soya crop as a post-emergence or pre-emergence treatment.

33. A method according to claim 31, wherein the crop is a cereal crop infested or susceptible to infestation by at least one dicotyledon weed.

34. A method according to claim 33 in which the dicotyledon weed is abutilon, xanthium or ipomea.

35. A method according to any one of claims 31 to 34 wherein the compound of the formula (I) is applied at a rate of 0.05 to 5 kg/ha.

36. A method according to any one of claims 31 to 35, wherein the compound of the formula (I) is applied at a rate of 0.1 to 2 kg/ha.

37. A method according to claim 31 substantially as hereinbefore described.

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